

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 10-289813

(43)Date of publication of application : 27.10.1998

(51)Int.Cl.

H01F 1/053  
C22C 38/00

(21)Application number : 09-098613

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(22)Date of filing : 16.04.1997

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## (54) RARE-EARTH MAGNET

## (57)Abstract:

PROBLEM TO BE SOLVED: To inhibit the oxidization of rare-earth elements being contained in an R-T-B rare-earth magnet alloy and to reduce the B-rich phase of a rare-earth magnet while an increase in the coarseness of the R<sub>2</sub>T<sub>14</sub>B main phase of the magnet is suppressed by a method wherein the magnet has a composition consisting of R, B, Co, M, oxygen, nitrogen and carbon, which respectively have a specified wt.%, with the balance of Fe, the coercive force of the magnet at a specified temperature is specified and at the same time, the area ratio of the B-rich phase is specified.

SOLUTION: A rare-earth magnet has a composition consisting of 27.0 to 31.0 wt.% of R (The R is one kind of the element or more than two kinds of the elements out of rare-earth elements including Y.), 0.8 to 1.02 wt.% of B, 0.5 to 50 wt.% of Co, 0.01 to 1.0 wt.% of M (The M is one kind of the element or more than two kinds of the elements out of Ga, Al and Cu), 0.25 wt.% or lower of oxygen, 0.02 to 0.15 wt.% of nitrogen and 0.15 wt.% or lower of carbon with the balance of Fe. Moreover, the coercive force of the magnet at 20° C is 13 kOe or higher and at the same time, the area ratio of the B-rich phase of the magnet is 0 to 0.5% and the area ratio of main phase crystal grains of a grain diameter of 16 μm or wider is 10% or lower.

## LEGAL STATUS

[Date of request for examination] 06.12.2000

[Date of sending the examiner's decision of rejection] 08.11.2002

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection] 2002-23582

[Date of requesting appeal against examiner's decision of rejection] 06.12.2002

[Date of extinction of right]

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(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開平10-289813

(43) 公開日 平成10年(1998)10月27日

(51) Int.Cl. <sup>6</sup>	識別記号	F I	
H 0 1 F 1/053		H 0 1 F 1/04	H
C 2 2 C 38/00	3 0 3	C 2 2 C 38/00	3 0 3 D

審査請求 未請求 請求項の数 3 O L (全 7 頁)

(21) 出願番号 特願平9-98613

(22) 出願日 平成9年(1997)4月16日

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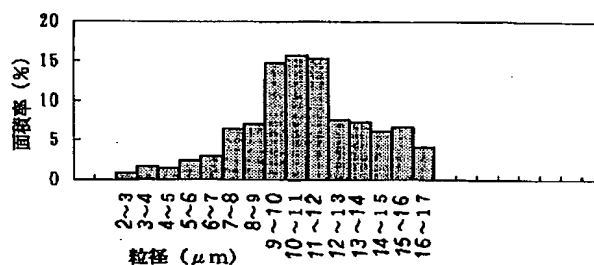
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(54) 【発明の名称】 希土類磁石

(57) 【要約】

【課題】 従来に比べてR-T-B系希土類磁石合金に含有される希土類元素の酸化を抑制し、かつR2T14B主相の粗大化を抑えながらBリッチ相を低減することでR2T14B主相の体積率を高めて高い磁気特性を得られるようにしたR-T-B系希土類焼結磁石を提供する。

【解決手段】 Rを27.0～31.0wt% (RはYを含む希土類元素のうちの1種または2種以上)、Bを0.8～1.02wt%、Coを0.5～5.0wt%、Mを0.01～1.0wt% (MはGa、Al、Cuのうちの1種または2種以上)、酸素を0.25wt%以下、窒素を0.02～0.15wt%、炭素を0.15wt%以下、残部Feからなる組成を有し、20℃における保磁力が13kOe以上であるとともに、Bリッチ相の面積率が0～0.5%であることを特徴とする希土類磁石。



## 【特許請求の範囲】

【請求項1】 Rを27.0～31.0wt%（RはYを含む希土類元素のうちの1種または2種以上）、Bを0.8～1.02wt%、Coを0.5～5.0wt%、Mを0.01～1.0wt%（MはGa、Al、Cuのうちの1種または2種以上）、酸素を0.25wt%以下、窒素を0.02～0.15wt%、炭素を0.15wt%以下、残部Feからなる組成を有し、20℃における保磁力が13kOe以上であるとともに、Bリッチ相の面積率が0～0.5%であることを特徴とする希土類磁石。

【請求項2】 結晶粒径が16μm以上である主相結晶粒の面積率が10%以下であることを特徴とする請求項1に記載の希土類磁石。

【請求項3】 Dy濃度が、主相結晶粒の粒界近傍が高く、主相結晶粒の中心部で低いことを特徴とする請求項1または2に記載の希土類磁石。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】 本発明は、従来に比べて非磁性相を低減させるとともに主相結晶粒の粗大化を抑えることにより高い磁気特性を得られるようにしたR-T-B系希土類磁石（TはFeまたはFeCoの混合物）に関する。

## 【0002】

【従来の技術】 資源的に比較的豊富で安価なNdおよびFeを主成分とするNd-Fe-B系希土類永久磁石は、非常に優れた磁気特性を有することから年々需要が増し、近年では希土類磁石市場の大半を占めるようになった。これに伴いその研究開発も盛んであり、とくに組成の異なる2つの金属粉末を混合して用いる方法（以下、ブレンド法という。）は、その2つの金属粉末中の相を制御することにより高い磁気特性が得られることから多くの方法が提案されている。ブレンド法では組成の異なる2つの合金を混合するが、主相であるR<sub>2</sub>T<sub>14</sub>B相（RはYを含む希土類元素のうちの少なくとも1種以上、TはFeまたはFeCoの混合物）に近い組成を有する主相形成用合金と、Rリッチな第2相形成用合金とを混合する方法が多用されており、例えば特開昭63-93841ではR<sub>2</sub>T<sub>14</sub>B相とそれよりもRリッチなR-X合金（XはFeまたはFeとB、Al、Ti、V、Co、Zr、Nb、Moのうちの少なくとも1種以上の混合物）からなる溶湯物の急冷によって得られる合金を混合して、磁石合金を製造する方法が提案されている。また例えば特開平5-175026、特開平5-175027などでは、R<sub>2</sub>T<sub>14</sub>B相を主体とする合金粉末と、MgCu<sub>2</sub>型、PuNi<sub>3</sub>型、CaZn<sub>5</sub>型などの結晶型を有する金属間化合物粉末を混合し成形、焼結することによって磁石合金を得ている。これらの方法では、組成の異なる2つの合金を用いることによって、粉碎性、焼

結性さらには磁石合金中の組織を改善し、単一合金を出発原料とする場合（以下、シングル法という。）に比べて磁気特性を向上できることが報告されている。

## 【0003】

【発明が解決しようとする課題】 上記ブレンド法において、磁石合金として実用上有効な保磁力を得るためには主相形成用合金、第2相形成用合金を2～10μm程度の微粉に粉碎する必要があるが、通常の方法ではこの際に激しい酸化を生じ、発火の危険性すらある。酸化された含有希土類元素は焼結時に液相としての役割を果たさず、希土類磁石合金の密度の向上に寄与しない。そのため従来の方法では酸化される含有希土類元素量を予想し、希土類磁石の原料合金にあらかじめ必要量より多い希土類元素を含ませる必要があった。また、希土類酸化物は焼結後の希土類磁石合金中に残存し、有効な磁化を発現する主相たるR<sub>2</sub>T<sub>14</sub>B相の体積率を下げるため、従来は希土類磁石合金の持つポテンシャルに比べて小さな残留磁束密度しか得られなかった。また、希土類焼結磁石中の主相結晶粒径を微細化するためにNb等の非磁性元素を添加しなければならず、このNb添加もまた残留磁束密度を低下させる要因となっている。さらに従来の希土類磁石合金中には上記希土類酸化物の他にも磁性に寄与しない不純物相が存在し、R<sub>2</sub>T<sub>14</sub>B相の体積率を下げるため残留磁束密度をより小さくしている。したがって、本発明の課題は、従来に比べてR-T-B系希土類磁石合金に含有される希土類元素の酸化を抑制し、かつR<sub>2</sub>T<sub>14</sub>B主相の粗大化を抑えながらBリッチ相を低減することでR<sub>2</sub>T<sub>14</sub>B主相の体積率を高めて高い磁気特性を得られるようにしたR-T-B系希土類焼結磁石を提供することである。また、本発明はNb無添加でも微細な結晶粒径のR<sub>2</sub>T<sub>14</sub>B主相が得られ、さらにR<sub>2</sub>T<sub>14</sub>B主相におけるDy元素の分布を制御することによって従来より高い磁気特性の得られるR-T-B系希土類焼結磁石を提供することである。

## 【0004】

【課題を解決するための手段】 本発明者らは、R-T-B系希土類磁石合金の微粉碎時の酸化を極力抑えることが可能な後述の湿式プロセスとブレンド法とを組み合わせ、微細なR<sub>2</sub>T<sub>14</sub>B主相の体積率を従来より増大させるためのR-T-B系希土類焼結磁石の合金設計を鋭意検討した結果、下記の本発明に想到した。上記課題を解決した本発明は、Rを27.0～31.0wt%（RはYを含む希土類元素のうちの1種または2種以上）、Bを0.8～1.02wt%、Coを0.5～5.0wt%、Mを0.01～1.0wt%（MはGa、Al、Cuのうちの1種または2種以上）、酸素を0.25wt%以下、窒素を0.02～0.15wt%、炭素を0.15wt%以下、残部Feからなる組成を有し、20℃における保磁力が13kOe以上であるとともに、Bリッチ相の面積率が0～0.5%であることを特徴とする

希土類磁石である。本発明では、結晶粒径が $16\mu\text{m}$ 以上である主相結晶粒の面積率が $10\%$ 以下であることが高い磁気特性を得るために好ましく、結晶粒径が $14\mu\text{m}$ 未満である主相結晶粒の面積率が $80\%$ 以上であるとともに結晶粒径が $16\mu\text{m}$ 以上である主相結晶粒の面積率が $10\%$ 以下であることがより好ましく、結晶粒径が $14\mu\text{m}$ 未満である主相結晶粒の面積率が $80\%$ 以上であるとともに結晶粒径が $16\mu\text{m}$ 以上である主相結晶粒の面積率が $5\%$ 以下であることが特に好ましい。また、Dy濃度が、主相結晶粒の粒界近傍で高く、主相結晶粒の中心部で低い場合に高い磁気特性が得られている。

【0005】本発明によれば湿式成形プロセスとブレンド法とを組み合わせるという独創的な概念をさらに発展させた適切なR-T-B系希土類磁石の合金設計により、ブレンド法の真のポテンシャルを発現するR-T-B系希土類磁石組成を発明することができた。本発明で採用したブレンド法は組成の異なるA合金およびB合金を用いる。A合金およびB合金はアーク溶解、高周波溶解などで鑄造してもよいし、溶湯を急冷して得られる $0.1\sim0.4\text{mm}$ の薄板状合金（以下ストリップキャスト合金という。）を用いてもよいが、A合金はストリップキャスト合金を、またB合金は鑄造合金を用いることが好ましい。A合金をストリップキャスト合金とすると $\text{R}_2\text{Fe}_{14}\text{B}$ 主相の間に細かくRリッチ相が分散された組織が得られる。このRリッチ相は水素処理などにより脆化され、微粉砕時にはその脆化部分より粉砕が進むため粉砕性が向上し焼結後に微細な主相結晶粒径が得られるようになる。また、B合金を鑄造材として、適切な融点の高い合金相を析出させることによって焼結時に粗大な結晶粒ができるのを防ぐことができる。

【0006】A合金は主として $\text{R}_2\text{Fe}_{14}\text{B}$ 相からなる。A合金はR量が $26.7\sim32.0\text{wt}\%$ 、B量が $0.9\sim2.0\text{wt}\%$ 、M（MはGa、Al、Cuのうち少なくとも1種以上）量が $0.1\sim3.0\text{wt}\%$ 、残部Feからなる組成のものが好ましい。A合金中のR量は $26.7\sim32.0\text{wt}\%$ が好ましい。R量が $26.7\text{wt}\%$ 未満であると $\text{R}_2\text{Fe}_{14}\text{B}$ 相の生成が十分ではなく軟磁性を持つ $\alpha\text{-Fe}$ などが析出し、また $32.0\text{wt}\%$ を越えるとRを多量に含むRリッチ相が増加し酸素量が増える結果、いずれも磁気特性が低下する。また、A合金中のB量は $0.9\sim2.0\text{wt}\%$ が望ましい。B量が $0.9\text{wt}\%$ 未満であると $\text{R}_2\text{Fe}_{14}\text{B}$ 相の生成が十分ではなく、軟磁性体である $\text{R}_2\text{Fe}_{17}$ 相が析出し、磁気特性を低下させる要因となる。さらに、A合金中のMの量は $0.1\sim3.0\text{wt}\%$ が好ましい。M元素は保磁力の向上に寄与するが、 $0.1\text{wt}\%$ 未満では十分な保磁力が得られず、 $3.0\text{wt}\%$ を越える添加では残留磁束密度が低下する。このようにして作製した合金Aは $800\sim1200^\circ\text{C}$ の温度範囲で $1\sim50$ 時間熱処理し、 $\alpha\text{-Fe}$ や $\text{R}_2\text{Fe}_{17}$ 相などを低減することが

好ましい。

【0007】B合金はA合金よりもR量が多く、主には $\text{R}_1\text{T}_2$ 相、 $\text{R}_1\text{T}_3$ 相、 $\text{R}_2\text{T}_{17}$ 相（TはFeおよび/またはCo）などにより構成される。B合金はR量が $35\sim70\text{wt}\%$ 、Co量が $5\sim50\text{wt}\%$ 、M量が $0.1\sim3.0\text{wt}\%$ 、残部Feからなる組成のものが好ましい。B合金のR含有量は $35\sim70\text{wt}\%$ とするのがよい。R量が $35\text{wt}\%$ 未満では $\alpha\text{-Fe}$ などの軟磁性を持つ相が生じる。またR量が $70\text{wt}\%$ を越えるとB合金中にRを多量に含むRリッチ相を生成して粉砕時に非常に酸化され易くなり、さらに焼結時には低温でそのRリッチ相が溶解するため異常粒成長の原因にもなる。ここで、R成分としてDyを添加する場合には、DyはA合金よりもB合金に多く配合することが望ましい。B合金に配合されたDyは焼結時に主相結晶粒内に拡散して行く結果、主相結晶粒においてDy濃度が粒界近傍で高く中心部で低い組織が得られる。したがって、Dy濃度の低い主相結晶粒の中央部で高い残留磁束密度が得られ、Dy濃度の高い主相結晶粒の粒界付近で逆磁場が印加された際に逆磁区ができるのを阻止するため高い保磁力が得られるものと判断される。また、B合金中のCo添加量は $5\sim50\text{wt}\%$ とするのがよい。CoはB合金に添加することが好ましく酸化され易いB合金の耐酸化性を向上させる作用がある。さらに、CoはR-Fe-B系希土類焼結磁石中の粒界に含有され耐食性の向上に寄与する他、主相中に拡散しキュリー点を上げて耐熱性を向上させる作用があるが、B合金への添加量が $5\text{wt}\%$ 未満ではこれらの作用効果が不十分であり、また $50\text{wt}\%$ より多い添加では飽和磁束密度が顕著に低下する。B合金のM含有量は $0.1\sim3.0\text{wt}\%$ がよい。 $0.1\text{wt}\%$ 未満では十分な保磁力が得られず、 $3.0\text{wt}\%$ を越えて添加すると残留磁束密度が顕著に低下するので好ましくない。

【0008】A合金およびB合金は水素処理、バンタムミルなどにより粗粉砕を行い、粗粉とする。次いでA合金粗粉 $99\sim70\text{wt}\%$ に対してB合金粗粉 $1\sim30\text{wt}\%$ をV型混合機、ボールミルなどにより均一化混合する。このときB合金粗粉が $1\text{wt}\%$ 未満では焼結性が悪くなり、焼結体密度が上がらないため十分な磁石特性が得られない。また、 $30\text{wt}\%$ より多い場合にはRが過多となるため、小さな残留磁束密度しか得られない。混合後の粗粉はRを $27\sim31\text{wt}\%$ 、Bを $0.5\sim2.0\text{wt}\%$ 、Coを $0.5\sim5\text{wt}\%$ 、Mを $0.01\sim1.0\text{wt}\%$ 含むようにするとよい。混合後の粗粉のRが $27\text{wt}\%$ 未満では焼結に必要な液相が得られず低い焼結体密度となるため、磁気特性は低い。Rが $31\text{wt}\%$ より多い場合ではRが過多となるため小さな残留磁束密度しか得られない。Bは $0.5\text{wt}\%$ 未満では $\text{R}_2\text{T}_{14}\text{B}$ 相が十分形成されず、 $2.0\text{wt}\%$ より多い場合には非磁性のBリッチ相を生じるため、いずれも小さな残

留磁束密度しか得られない。Co量は0.5wt%未満では耐食性、耐熱性が十分ではなく、5wt%より多い場合には残留磁束密度を大きく下げる。さらに、Mは0.01wt%未満では十分な保磁力が得られず、1.0wt%より多い場合には $R_2T_{14}B$ 相の体積率が下がるため残留磁束密度が低下する。

【0009】このようにして得られたA合金粗粉とB合金粗粉とを混合した原料粗粉は例えばジェットミルなどの粉砕機によって含有酸素量が実質的に0wt%の不活性ガス雰囲気中にて微粉砕を行い、平均粒径（空気透過法による）2～10 $\mu$ m程度の微粉とする。微粉の回収にあたっては、ジェットミルなどの微粉回収口に鉱物油、植物油、合成油等の微粉の酸化の進行を阻止可能な特殊な溶媒を満たした容器を設置し、不活性ガス雰囲気中で直接微粉をその特殊溶媒中に回収しスラリー化する。こうして得たスラリー状の原料を所定の磁場異方性の付与ができる金型を用いて磁場中で湿式成形し、成形体とする。スラリーまたは成形体状態では特殊溶媒中に前記微粉末粒子が保持されて酸素と隔絶されているので微粉または成形体の酸素量は増加しない。次にこの成形体を例えば $1 \times 10^{-2}$  torr以上の真空炉で100～300℃程度に加熱し、成形体内の含有溶媒を十分に除去する。次いで引き続き、同一真空炉の温度を1000～1200℃程度まで引き上げ、例えば $1 \times 10^{-3} \sim 9 \times 10^{-6}$  torrの真空度下で焼結を行う。次いで、その焼結体を焼結温度未満の温度で例えば2段階あるいは3段階の熱処理をすることによって得られる本発明の希土類焼結磁石は、R:27.0～31.0wt%、B:0.8～1.02wt%、Co:0.5～5.0wt%、M:0.01～1.0wt%、含有酸素量が0.25wt%以下、含有窒素量が0.02～0.15wt%、含有炭素量が0.15wt%以下、残部がFeの組成を有している。R、B、Co、Feの量は粗粉原料の組成調整によるものである。含有される酸素、窒素、炭素は原料合金中に不純物として含有されていたり、粗粉砕時や加熱時に大気や特殊溶媒等から混入するものであるが、これら不可避不純物はR-T-B系希土類磁石合金中でNd<sub>2</sub>O<sub>3</sub>、Nd<sub>2</sub>C<sub>3</sub>、NdN等の非磁性化合物を作り主相体積率を下げ、さらに焼結時に液相として働くRの量を減少させて焼結を阻害するため、含有酸素量は0.25wt%以下、含有窒素量は0.02～0.15wt%、含有炭素量は0.15wt%以下としなければならない。この組成範囲にあるときに本発明の高い磁気特性を有したR-T-B系希土類焼結磁石を得ることができる。

【0010】本発明の希土類磁石が高い磁気特性を有している理由を説明する。本発明においては湿式回収、湿式成形を行うことにより微粉砕以降の酸化を防ぐことができる。例えば本発明のR-T-B系希土類磁石は例えば含有酸素量が実質的に0%の不活性ガス中で微粉砕

し、さらに不活性ガス雰囲気中において微粉の酸化を阻止可能な特殊な溶媒中に前記微粉を直接回収する。さらにこの特殊溶媒の共存下で磁場中成形し、得られた成形体を十分に脱溶媒後焼結することで含有酸素量、含有窒素量、含有炭素量を上記範囲に安定して抑えることができる。さらにはA合金、B合金の組成および組織をこの低酸素法に対し最適なものとして設定したため焼結性等を改善でき、従来より多用されているNbなどの粒成長を防ぐ非磁性元素を添加しなくても希土類磁石合金中の組織を最良のものとすることができる結果、微細な主相比率を従来に比べて高めることが可能となった。さらに、Co、Dyなどの添加元素をB合金に添加し、主相結晶粒内におけるDy元素の分布を制御できるようになった。すなわち、低酸素法によりブレンド法の持つポテンシャルを十分引き出すことによって、低酸素量で磁気特性に優れたR-T-B系希土類磁石合金を得ることが可能となった。なお、上述の酸素濃度が実質的に0wt%である不活性ガスとは、例えばR-Fe-B系原料粗粉を10kg/Hr程度で微粉砕できる能力を有する生産型のジェットミル粉砕機の場合では、不活性ガス中の酸素濃度が0.01vol%以下、より好ましくは0.005vol%以下、特に好ましくは0.002vol%以下の不活性ガスをいう。

#### 【0011】

【発明の実施の形態】以下、本発明を実施例をもって具体的に説明するが、本発明の内容はこれによって限定されるものではない。

#### 【0012】（実施例1）重量百分率でNd27.5

%、Pr0.4%、B1.05%、Ga0.1%、Cu0.1%、残部Feからなる合金Aを作製するため、純度95%以上のNd、Pr、B、Ga、Cu、Feを所定量秤量し、Arガス雰囲気中で高周波溶解し、この合金溶湯を同じくArガス雰囲気中で銅製単ロールに注湯し、いわゆるストリップキャスト法で薄板状の合金とした。この合金の板厚は0.1～0.3mmであった。さらに合金Aを真空炉中に装入し、 $5 \sim 8 \times 10^{-2}$  Torrの条件下で1000℃×4時間熱処理をした。また、重量百分率でNd31.5%、Pr0.5%、Dy15.0%、Al0.2%、Co20.0%、残部Feからなる合金Bを作製するために純度95%以上のNd、Pr、Dy、Al、Co、FeをArガス雰囲気中で高周波溶解し、铸造した。このようにして得られた合金Aおよび合金Bは、空気を排除した炉内で水素を吸蔵させた後、真空排気を行いながら500℃まで加熱した後、冷却し、その後粗粉砕して32メッシュアンダーの粗粉とした。次に、合金Aの粗粉90重量部に対して合金Bの粗粉10重量部を秤量後V型混合機にて均一化混合し、原料粗粉とした。この原料粗粉を、N<sub>2</sub>ガス置換して酸素濃度を0.001vol%としたジェットミル内に装入してジェットミル粉砕した。ジェットミルの粉砕微粉回収口

には鉍物油（出光興産製、商品名出光スーパーゾルPA-30）を満たした容器を設置し、N<sub>2</sub>ガス雰囲気中において微粉を直接鉍物油中に回収してスラリー化した。微粉の平均粒径（空気透過法）は4.5 μmであった。次に、このスラリーを所定の磁場金型のキャビティ内に10.5 kOeの磁場を印加しながら注入し1.0 ton/cm<sup>2</sup>の成形圧で湿式成形した。配向磁場の印加方法は、成形方向と垂直である。次に得られた成形体を5~7×10<sup>-2</sup> torrの真空中で200℃×1時間加熱して含有鉍物油を十分に除去した後、続いて同一真空炉で5~8×10<sup>-5</sup> torrの真空中で15℃/分の昇温速度で1070℃まで昇温し、その温度で2時間保持して焼結し冷却した。次に、この焼結体にAr雰囲気中で900℃×2時間と480℃×1時間の熱処理を各1回行って本発明の希土類磁石を得た。この希土類磁石の代表的な組成は重量百分率でNd 27.7%、Pr 0.6%、Dy 1.5%、B 0.92%、Co 2.1%、Al 0.1%、Ga 0.1%、Cu 0.1%、含有酸素量0.17%、含有窒素量0.05%、含有炭素量0.07%、残部Feであった。またこの希土類磁石の20℃における代表的な磁気特性は、表1に示す通りBr=14.2 kG、iHc=14.9 kOe、(BH)<sub>max</sub>=48.4 MGOeという良好な磁気特性である。また、得られた希土類磁石の任意のもののマイクロ組織を光学顕微鏡で観察したが、ブリッチ相は観察されなかった。また、観察した視野のR<sub>2</sub>T<sub>14</sub>B主相結晶粒の総面積に対し、図1の横軸に示す粒径範囲にある各R<sub>2</sub>T<sub>14</sub>B主相の面積をその総面積で除した面積率(%)を図1の縦軸にとってこの実施例1で得られた希土類磁石を評価した。ここで、例えば図1の横軸にとっている粒径(μm)の16~17というのは結晶粒径が16 μm以上でかつ17 μm未満の範囲にあるR<sub>2</sub>T<sub>14</sub>B主相を示している。また、図1におけるR<sub>2</sub>T<sub>14</sub>B主相の結晶粒径、面積率は下記のようにして測定、評価した。まず、型式ニコンUFX-11の光学顕微鏡を用いて、上記のマイクロ観察試料の任意部分の光学顕微鏡写真(倍率1000倍)を撮影し、次いでこの光学顕微鏡写真をプラネトロン社製の画像処理ソフト(Image pro. plus (DOS/V))がインストールされている所定のパーソナルコンピュータに備えられたスキャナから読み込み、画像処理した。この画像処理で測定した任意の各主相結晶粒の面積を(S<sub>i</sub>)として、さらに各主相結晶粒の観察断面形状を円と仮定し、各主相結晶粒径(d<sub>i</sub>)=(4×S<sub>i</sub>÷π)<sup>1/2</sup>で定義した。また、主相の面積率は観察した対象の主相結晶粒の全面積(S<sub>total</sub>)に対して図1の横軸に示す粒径範囲にある主相結晶粒の面積(S<sub>i</sub>')の比率、すなわち(面積率)=S<sub>i</sub>'÷S<sub>total</sub>×100(%)で定義した。評価結果は図1に示すように、実施例1のものはR<sub>2</sub>T<sub>14</sub>B主相における結晶粒径14 μm未満の結晶粒の面積率は83.2%、結晶粒径16 μm以上の結晶粒

の面積率は4.0%であった。また、得られた希土類磁石の任意のもののFe、Nd、Dy、B含有元素の挙動をEPMAにより分析した結果を図2に示す。図2においてNdの極大ピーク間が主相結晶粒であるが、この主相結晶粒においてDy濃度に勾配があることがわかる。主相結晶粒の粒界付近ではDy濃度が高く、主相結晶粒の中心部でDy濃度が低い傾向にある。これはDyをB合金にのみ添加したためであると考えられる。このDy分布の偏在によって、主相結晶粒の中心部が高いBrを担い主相結晶粒の粒界付近が高いiHcを担っているものと推定される。

【0013】(実施例2)重量百分率でNd 27.5%、Pr 0.4%、B 1.11%、Ga 0.1%、Cu 0.1%、残部Feからなる合金Aを用いた以外は実施例1と同様にして本発明のR-T-B系異方性焼結希土類磁石を製作した。得られた希土類磁石の代表的な組成分析値は重量百分率でNd 27.8%、Pr 0.5%、Dy 1.5%、B 1.00%、Co 2.2%、Al 0.1%、Ga 0.1%、Cu 0.1%、含有酸素量0.20%、含有窒素量0.04%、含有炭素量0.07%、残部Feであった。また、得られた希土類磁石の代表的な磁気特性を20℃において測定したところ、表1に示すようにBr=14.1 kG、iHc=15.0 kOe、(BH)<sub>max</sub>=47.9 MGOeという良好な磁気特性を得た。また、この実施例2の希土類磁石の任意のもののマイクロ組織を観察したところ、わずかにブリッチ相が存在していることが光学顕微鏡で確認できたが、観察面の総面積に対するブリッチ相の面積率は0.36%と小さかった。

【0014】(比較例1)重量百分率でNd 27.5%、Pr 0.4%、B 1.17%、Ga 0.1%、Cu 0.1%、残部Feからなる合金A、および重量百分率でNd 31.5%、Pr 0.5%、Dy 15.0%、Al 0.2%、Co 20.0%、残部Feからなる合金Bを用いた以外は実施例1と同様にしてR-T-B系異方性焼結磁石を作製した。得られた希土類磁石の代表的な組成は重量百分率でNd 27.9%、Pr 0.5%、Dy 1.5%、B 1.08%、Co 2.1%、Al 0.1%、Ga 0.1%、Cu 0.1%、含有酸素量0.19%、含有窒素量0.05%、含有炭素量0.07%、残部Feであり、実施例1、2に比べてB含有量が高い。得られた希土類磁石の代表的な磁気特性を20℃において測定したところ、表1に示すようにBr=13.9 kG、iHc=15.2 kOe、(BH)<sub>max</sub>=46.9 MGOeとなり、Brおよび(BH)<sub>max</sub>が実施例1、2よりも低かった。また、得られた希土類磁石の任意のもののマイクロ組織を実施例2と同様にして評価したところ、ブリッチ相の面積率は0.77%であり、実施例2の約2倍である。以上のことから、比較例1のものは実施例1、2に比べてB含有量が多いため、非磁性のブリッチ相が多く形成されて相対的にR<sub>2</sub>T<sub>14</sub>B主相の体積率が低下し

たため、 $B_r$  および  $(BH)_{max}$  が低下していると考えられる。

【0015】（比較例2）シングル法にて比較例1と同一組成の希土類磁石合金を作製した。純度95%以上のNd、Pr、Dy、B、Al、Ga、Cu、Co、Feを所定量秤量し、実施例1と同様のストリップキャスト法にて重量百分率でNd 27.9%、Pr 0.45%、Dy 1.5%、B 1.05%、Co 2.0%、Al 0.15%、Ga 0.08%、Cu 0.10%、残部Feの組成を有する原料合金を得た。このようにして得られた原料合金は、空気を排除した炉内で水素を吸蔵させた後、真空排気を行いながら500℃まで加熱した後、冷却し、その後粗粉碎して32メッシュアンダーの原料粗粉とした。この原料粗粉を、 $N_2$ ガスにて置換して酸素濃度を0.001vol%としたジェットミル内に装入してジェットミル粉碎し、微粉回収口には鉱物油（出光興産製、商品名出光スーパーゾルPA-30）を満たした容器を設置し、 $N_2$ ガス雰囲気中にてジェットミル粉碎した微粉を鉱物油中に直接回収しスラリー化した。微粉の平均粒径（空気透過法）は4.4 $\mu m$ であった。以降は実施例1と同様にしてR-T-B系異方性焼結磁石を得た。このものの代表的な組成分析値は重量百分率でNd 27.7%、Pr 0.5%、Dy 1.5%、B 1.06%、Co 2.0%、Al 0.1%、Ga 0.1%、Cu 0.1%、含有酸素量0.16%、含有窒素量0.05%、含有炭素量0.07%、残部Feであった。また、得られた上記希土類磁石の磁気特性を20℃において測定したところ、表1に示すように $B_r = 13.8 kG$ 、 $iH_c = 14.1 kOe$ 、 $(BH)_{max} = 46.4 MGOe$ であり、実施例1、2に比べて低い磁気特性を得た。また、得られた希土類磁石から任意のものを選んで、実施例1と同様にして各結晶粒径範囲にある主相結晶粒の面積率を評価した結果を図3に示した。図3より、図1（実施例1）に比べて主相の結晶粒径分布が特に大粒径側にブロードであり、粒径16 $\mu m$ 未満の主相結晶粒の面積率は83.6%（したがって16 $\mu m$ 以上は16.4%）、粒径19 $\mu m$ 以上の主相結晶粒の面積率は6.7%であった。このように、シングル法では上記本発明のブレンド法によるものに比べて主相結晶粒が粗大化し易く、主相の結晶粒径のばらつきが大きくブロードな粒径分布となっていることから、シングル法では主相結晶粒成長の抑制が不十分であり、ブレンド法で作製した本発明のものよりも磁気特性が低下したと判定される。

【0016】（比較例3）シングル法にてNb添加のR-T-B系希土類磁石合金を作製した。純度95%以上のNd、Pr、B、Nb、Ga、Cu、Co、Feを所定量秤量し、実施例1と同様のストリップキャスト法にて重量百分率でNd 27.9%、Pr 0.45%、Dy 1.5%、B 0.90%、Co 2.0%、Nb 0.70%、Al 0.15%、Ga 0.08%、Cu 0.10%、残部F

eの組成を有する原料合金を得た。この合金には粒成長を抑制するためにNbが添加されている。以降は実施例1と同様にして粉碎、湿式回収、湿式成形、脱溶媒、焼結、熱処理を行いNb添加のR-T-B系希土類焼結磁石を作製した。この磁石の代表的な組成分析値は重量百分率でNd 27.7%、Pr 0.57%、Dy 1.5%、B 0.87%、Co 2.0%、Nb 0.72%、Al 0.10%、Ga 0.07%、Cu 0.12%、含有酸素量0.13%、含有窒素量0.08%、含有炭素量0.07%、残部Feであった。また、得られた希土類磁石の代表的な20℃における磁気特性は、表1に示す通り $B_r = 13.3 kG$ 、 $iH_c = 16.1 kOe$ 、 $(BH)_{max} = 42.7 MGOe$ であり、実施例1、2より低かった。

【0017】（比較例4）シングル法にてNb添加のR-T-B系希土類磁石合金を作製した。純度95%以上のNd、Pr、B、Nb、Ga、Cu、Co、Feを所定量秤量し、実施例1と同様の条件のストリップキャスト法にて重量百分率でNd 27.9%、Pr 0.45%、Dy 1.5%、B 1.10%、Co 2.0%、Nb 0.70%、Al 0.15%、Ga 0.08%、Cu 0.10%、残部Feの組成を有する原料合金を得た。この合金には粒成長を抑制するためにNbが添加されている。以降は実施例1と同様にして粉碎、湿式回収、湿式成形、脱溶媒、焼結、熱処理を行いR-T-B系希土類焼結磁石を作製した。この磁石の代表的な組成分析値は重量百分率でNd 27.8%、Pr 0.57%、Dy 1.5%、B 1.03%、Co 2.1%、Nb 0.71%、Al 0.14%、Ga 0.07%、Cu 0.12%、含有酸素量0.12%、含有窒素量0.06%、含有炭素量0.08%、残部Feであった。また、得られた希土類磁石の代表的な磁気特性は、表1に示す通り20℃において $B_r = 13.2 kG$ 、 $iH_c = 16.5 kOe$ 、 $(BH)_{max} = 42.0 MGOe$ であり実施例1、2よりも低磁気特性である。比較例3、4の結果からシングル法で作製したNb添加のR-T-B系希土類焼結磁石においては $B_r$ がB量に大きく依存しないことがわかった。また、この比較例4で得られた希土類磁石の任意のものについて含有されるFe、Nd、Dy、Bの挙動をEPMAにより分析した結果を図4に示す。図4では図1で観察されたような主相結晶粒におけるDyの濃度勾配は見られない。このことから実施例1で観察されたDyの濃度勾配はブレンド材に特有のものであることが確認された。

【0018】上記各実施例、各比較例で得られた磁気特性を表1にまとめた。

【表1】

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	$B_r(kG)$	$iH_c(kOe)$	$(BH)_m(MGOe)$
実施例1	14.2	14.9	48.4
実施例2	14.1	15.0	47.9
比較例1	13.9	15.2	46.9
比較例2	13.8	14.1	46.4
比較例3	13.3	16.1	42.7
比較例4	13.2	16.5	42.0

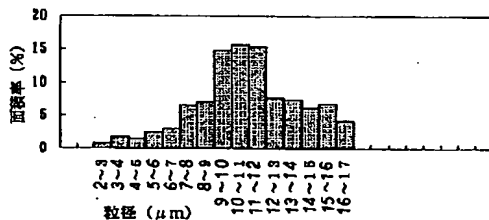
【0019】実施例1、2および比較例1で得られたBリッチ相の面積率を表2に示した。

【表2】

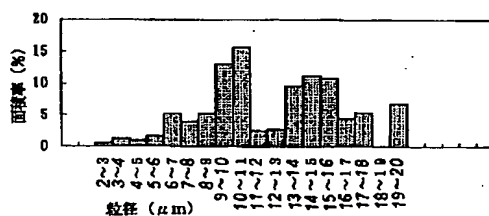
	Bリッチ相の面積率(%)
実施例1	0.00
実施例2	0.36
比較例1	0.77

【0020】

【図1】



【図3】



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【発明の効果】上記の通り、本発明の希土類磁石は、酸化物およびBリッチ相等の非磁性相を従来に比べて低減し主相体積率を高めるとともに主相の結晶粒成長を抑制したことで $B_r$ および $(BH)_{max}$ をR-T-B系磁石合金固有のポテンシャルに近づけることができた。さらには、Nb無添加でも主相の結晶粒径分布を微細化できるので、より非磁性相の体積率を低減可能である。このように、特長ある湿式プロセスとブレンド法とを組み合わせるとともに、その組み合わせに適したR-T-B系希土類磁石合金組成とした本発明の有用性は明らかである。

【図面の簡単な説明】

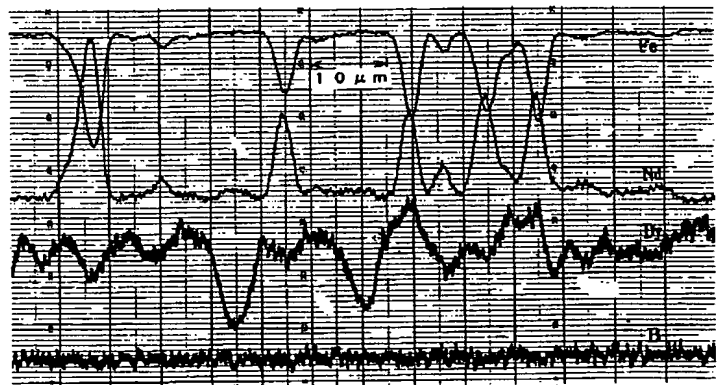
【図1】本発明の希土類磁石の主相結晶粒径分布と面積率との関係を示す図である。

【図2】本発明の希土類磁石のEPMA分析結果を示す図である。

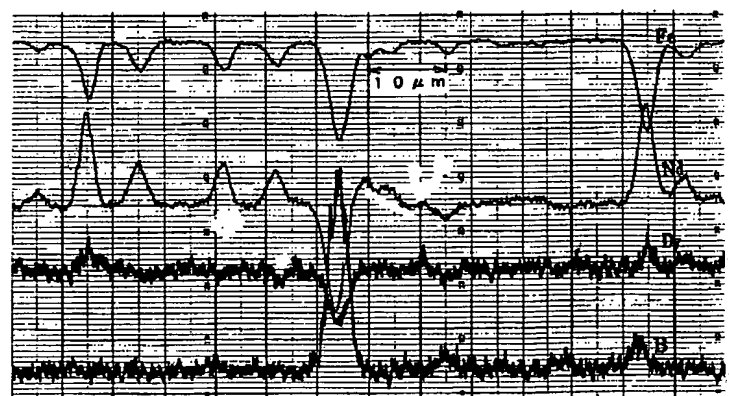
【図3】比較例の希土類磁石の主相結晶粒径分布と面積率との関係を示す図である。

【図4】比較例の希土類磁石のEPMA分析結果を示す図である。

【図2】



【図4】





# PATENT ABSTRACTS OF JAPAN

(11)Publication number : 10-289813

(43)Date of publication of application : 27.10.1998

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(51)Int.Cl. H01F 1/053

C22C 38/00

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(21)Application number : 09-098613 (71)Applicant : HITACHI METALS LTD

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(54) RARE-EARTH MAGNET

(57)Abstract:

PROBLEM TO BE SOLVED: To inhibit the oxidization of rare-earth elements being contained in an R-T-B rare-earth magnet alloy and to reduce the B-rich phase of a rare-earth magnet while an increase in the coarseness of the R<sub>2</sub>T<sub>14</sub>B main phase of the magnet is suppressed by a method wherein the magnet has a composition consisting of R, B, Co, M, oxygen, nitrogen and carbon, which respectively have a specified wt.%, with the balance of Fe, the coercive force of the magnet at a specified temperature is specified and at the same time, the area ratio of the B-rich phase is specified.

SOLUTION: A rare-earth magnet has a composition consisting of 27.0 to 31.0 wt.% of R (The R is one kind of the element or more than two kinds of the elements out of rare-earth elements including Y.), 0.8 to 1.02 wt.% of B, 0.5 to 50

wt.% of Co, 0.01 to 1.0 wt.% of M (The M is one kind of the element or more than two kinds of the elements out of Ga, Al and Cu), 0.25 wt.% or lower of oxygen, 0.02 to 0.15 wt.% of nitrogen and 0.15 wt.% or lower of carbon with the balance of Fe. Moreover, the coercive force of the magnet at 20°C is 13 kOe or higher and at the same time, the area ratio of the B-rich phase of the magnet is 0 to 0.5% and the area ratio of main phase crystal grains of a grain diameter of 16  $\mu\text{m}$  or wider is 10% or lower.

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## LEGAL STATUS

[Date of request for examination] 06.12.2000

[Date of sending the examiner's  
decision of rejection] 08.11.2002

[Kind of final disposal of application  
other than the examiner's decision of  
rejection or application converted  
registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's  
decision of rejection] 2002-23582

[Date of requesting appeal against  
examiner's decision of rejection] 06.12.2002

[Date of extinction of right]

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## CLAIMS

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[Claim(s)]

[Claim 1] R 27.0 - 31.0wt% (one sort in the rare earth elements in which R contains Y, or two sorts or more), They are 0.5 - 5.0wt% and M about 0.8 - 1.02wt% and Co in B 0.01 - 1.0wt% (it Ga(s) M) One sort in aluminum and Cu or two sorts or more, and oxygen Less than [ 0.25wt% ], The rare earth magnet characterized by the rate of area of B rich phase being 0 - 0.5% while it has the presentation which consists carbon of less than [ 0.15wt% ] and the remainder Fe 0.02 - 0.15wt% in nitrogen and the coercive force in 20 degrees C is 13 or more kOes.

[Claim 2] The rare earth magnet according to claim 1 characterized by the rate of area of the main phase crystal grain whose diameter of crystal grain is 16 micrometers or more being 10% or less.

[Claim 3] The rare earth magnet according to claim 1 or 2 with which Dy concentration is characterized by being high and being low in the core of the main phase crystal grain near the grain boundary of the main phase crystal grain.

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[Translation done.]

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## DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to the R-T-B system rare earth magnet (T is the mixture of Fe or FeCo) which enabled it to acquire high magnetic properties by suppressing big and rough-ization of the main phase crystal grain while reducing a nonmagnetic phase compared with the former.

[0002]

[Description of the Prior Art] Need came to occupy most rare earth magnet commercial scenes in increase and recent years every year from the Nd-Fe-B system rare earth permanent magnet which uses comparatively [ in resource ] abundant and cheap Nd and Fe as a principal component having the magnetic properties which were very excellent. In connection with this, the researches and developments are also prosperous, and since high magnetic properties are acquired when especially the approach (henceforth the blending method) of mixing two metal powder with which presentations differ, and using controls the phase in the two metal powder, many approaches are proposed. Although two alloys with which presentations differ are mixed by the blending method The alloy for the main phase formation which has the presentation near the R<sub>2</sub>T<sub>14</sub>B phase (at least one or more sorts in the rare earth elements in which R contains Y, and T are the mixture of Fe or FeCo) which is the main phase, R -- the approach of mixing the rich alloy for the 2nd phase formation uses it abundantly -

- having -- \*\*\*\* -- JP,63-93841,A -- R2T14 B phase and it -- R -- a rich R-X alloy (X -- Fe, or Fe and B --) The alloy obtained by quenching of the molten metal object which consists of at least one or more sorts of mixture of aluminum, Ti, V, Co, Zr, Nb, and the Mo is mixed, and the method of manufacturing a magnet alloy is proposed. Moreover, by JP,5-175026,A and JP,5-175027,A, the magnet alloy has been obtained, for example by mixing and sintering [ fabricate and ] the alloy powder which makes R2T14 B phase a subject, and the intermetallic-compound powder which has crystal molds, such as MgCu<sub>2</sub> mold, PuNi<sub>3</sub> mold, and CaZn<sub>5</sub> mold. By these approaches, by using two alloys with which presentations differ, the organization in a magnet alloy is improved to a grindability and a degree-of-sintering pan, and it is reported that magnetic properties can be improved compared with the case (henceforth the single method) where a single alloy is used as a start raw material.

[0003]

[Problem(s) to be Solved by the Invention] In order to acquire the practically effective coercive force as a magnet alloy, it is necessary to grind the alloy for the main phase formation, and the alloy for the 2nd phase formation to about 2-10-micrometer fines, and in the above-mentioned blending method, in this case, intense oxidation is produced and there is even danger of ignition by the usual approach. The content rare earth elements which oxidized do not play a role of the liquid phase at the time of sintering, and do not contribute to improvement in the consistency of a rare earth magnet alloy. Therefore, by the conventional approach, the amount of content rare earth elements which oxidizes needed to be expected, and many rare earth elements needed to be beforehand included in the raw material alloy of a rare earth magnet from the initial complement.

Moreover, the rare earth oxide remained in the rare earth magnet alloy after sintering, and in order to lower the rate of the volume of the main phase slack R2T14 B phase which discovers effective magnetization, compared with the potential which a rare earth magnet alloy has, only the small residual magnetic flux density was obtained conventionally. Moreover, in order to make detailed the

diameter of the main phase crystal grain in a rare earth sintered magnet, nonmagnetic elements, such as Nb, must be added, and it has become the factor in which this Nb addition also reduces a residual magnetic flux density. In the further conventional rare earth magnet alloy, the impurity phase which does not contribute to the magnetism other than the above-mentioned rare earth oxide exists, and in order to lower the rate of the volume of R2T14 B phase, the residual magnetic flux density is made smaller. Therefore, the technical problem of this invention is offering the R-T-B system rare earth sintered magnet which raises the rate of the volume of the R2T14B main phase by reducing B rich phase, and enabled it to acquire high magnetic properties, controlling oxidization of the rare earth elements contained in a R-T-B system rare earth magnet alloy compared with the former, and suppressing big and rough-ization of the R2T14B main phase. Moreover, this invention is offering the R-T-B system rare earth sintered magnet with which the R2T14B main phase of the diameter of crystal grain also with detailed Nb additive-free is obtained, and magnetic properties higher than before are acquired by controlling distribution of Dy element in the R2T14B main phase further.

[0004]

[Means for Solving the Problem] this invention persons hit on an idea to following this invention, as a result of examining wholeheartedly the alloy designing of the R-T-B system rare earth sintered magnet for increasing the rate of the volume of the detailed R2T14B main phase conventionally combining the below-mentioned wet process and the below-mentioned blending method which can suppress the oxidization at the time of pulverizing of a R-T-B system rare earth magnet alloy as much as possible. This invention which solved the above-mentioned technical problem R 27.0 - 31.0wt% (one sort in the rare earth elements in which R contains Y, or two sorts or more), They are 0.5 - 5.0wt% and M about 0.8 - 1.02wt% and Co in B 0.01 - 1.0wt% (it Ga(s) M) One sort in aluminum and Cu or two sorts or more, and oxygen Less than [ 0.25wt% ], While it has the presentation which consists carbon of less than [ 0.15wt% ] and the remainder

Fe 0.02 - 0.15wt% in nitrogen and the coercive force in 20 degrees C is 13 or more kOes, it is the rare earth magnet characterized by the rate of area of B rich phase being 0 - 0.5%. Are desirable in order to acquire magnetic properties with high the rate of area of the main phase crystal grain whose diameter of crystal grain is 16 micrometers or more being 10% or less in this invention. It is more desirable that the rate of area of the main phase crystal grain whose diameter of crystal grain is 16 micrometers or more while the rate of area of the main phase crystal grain whose diameter of crystal grain is less than 14 micrometers is 80% or more is 10% or less. While the rate of area of the main phase crystal grain whose diameter of crystal grain is less than 14 micrometers is 80% or more, it is desirable that the rate of area of the main phase crystal grain whose diameter of crystal grain is 16 micrometers or more is especially 5% or less. Moreover, Dy concentration is high near the grain boundary of the main phase crystal grain, and when low in the core of the main phase crystal grain, high magnetic properties are acquired.

[0005] According to this invention, by the alloy designing of a suitable R-T-B system rare earth magnet into which the original concept of combining a wet compaction process and the blending method was developed further, the R-T-B system rare earth magnet presentation which discovers the true potential of the blending method was able to be invented. The blending method adopted by this invention uses A alloy and B alloy with which presentations differ. although the 0.1-0.4mm sheet metal-like alloy (henceforth a strip cast alloy) which may cast A alloy and B alloy in the arc dissolution, the RF dissolution, etc., quenches a molten metal, and is obtained may be used -- A alloy -- a strip cast alloy -- moreover, as for B alloy, it is desirable to use a casting alloy. If A alloy is used as a strip cast alloy, the organization where R rich phase was finely distributed between the R<sub>2</sub>Fe<sub>14</sub>B main phases will be obtained. In order that this R rich phase may stiffen by hydrogen processing etc. and grinding may progress from that embrittlement part at the time of pulverizing, a grindability improves and the detailed diameter of the main phase crystal grain comes to be obtained after

sintering. Moreover, it can prevent making big and rough crystal grain into depositing an alloy phase with the suitable high melting point by making B alloy into a casting at the time of \*\*\*\* sintering.

[0006] A alloy mainly consists of R<sub>2</sub>Fe<sub>14</sub>B phase. A alloy has the desirable thing of the presentation which the amount of M (M is at least one or more sorts in Ga, aluminum, and Cu) becomes [ the amount of R / the amount of B ] from Remainder Fe. 0.1 - 3.0wt% 0.9 - 2.0wt% 26.7 - 32.0wt%. 26.7 - 32.0wt% of the amount of R in A alloy is desirable. If generation of R<sub>2</sub>Fe<sub>14</sub>B phase is not enough in the amount of R being less than [ 26.7wt% ], and alpha-Fe with soft magnetism etc. deposits and 32.0wt(s)% is exceeded, as a result of R rich phase which contains R so much increasing and the amount of oxygen increasing, magnetic properties fall [ all ]. Moreover, 0.9 - 2.0wt% of the amount of B in A alloy is desirable. Generation of R<sub>2</sub>Fe<sub>14</sub>B phase is not enough in the amount of B being less than [ 0.9wt% ], R<sub>2</sub>Fe<sub>17</sub> phase which is a soft magnetic material deposits, and it becomes the factor which reduces magnetic properties. Furthermore, 0.1 - 3.0wt% of the amount of M in A alloy is desirable. Although M element is contributed to improvement in coercive force, coercive force sufficient less than [ 0.1wt% ] is not acquired, but a residual magnetic flux density falls in addition exceeding 3.0wt%. Thus, it is desirable to heat-treat the produced alloy A in a 800-1200-degree C temperature requirement for 1 to 50 hours, and to reduce alpha-Fe and R<sub>2</sub>Fe<sub>17</sub> phase etc.

[0007] B alloy has many amounts of R, and is mainly constituted from an A alloy by R<sub>1</sub>T<sub>2</sub> phase, R<sub>1</sub>T three phase circuit, R<sub>2</sub>T<sub>17</sub> phase (T is Fe and/or Co), etc. B alloy has the desirable thing of the presentation which the amount of M becomes [ the amount of R / the amount of Co(es) ] from Remainder Fe 0.1 - 3.0wt% 5 - 50wt% 35 - 70wt%. R content of B alloy is good to consider as 35 - 70wt%. The phase in which the amount of R has soft magnetism, such as alpha-Fe, less than [ 35wt% ] arises. Moreover, since R rich phase which contains R so much in B alloy will be generated, it will become that it is very easy to oxidize at the time of grinding and the R rich phase will dissolve at low temperature further



at the time of sintering if the amount of R exceeds 70wt(s)%, it also becomes the cause of abnormality grain growth. Here, when adding Dy as an R component, as for Dy, blending into B alloy mostly is more desirable than A alloy. As a result of Dy blended with B alloy spreading and going in the main phase crystal grain at the time of sintering, in the main phase crystal grain, the organization in which Dy concentration is high near the grain boundary, and it is low in a core is obtained. Therefore, a high residual magnetic flux density is obtained in the center section of the main phase crystal grain with low Dy concentration, and when a reverse magnetic field is impressed near the grain boundary of the main phase crystal grain with high Dy concentration, in order to prevent that a reverse magnetic domain is made, it is judged as that from which high coercive force is acquired. Moreover, Co addition in B alloy is good to consider as 5 - 50wt%. Co has the operation which raises the oxidation resistance of B alloy with which adding into B alloy tends to oxidize preferably. Furthermore, although contain Co in the grain boundary in a R-Fe-B system rare earth sintered magnet, and it contributes to corrosion resistance improvement and also it has the operation which is spread in the main phase, gets the Curie point, and raises thermal resistance, by more addition than 50wt(s)%, saturation magnetic flux density falls [ the addition to B alloy ] notably insufficiently [ less than / 5wt% / such operation effectiveness ]. 0.1 - 3.0wt% of M content of B alloy is good. 0. Since a residual magnetic flux density will fall notably if coercive force sufficient less than [ 1wt% ] is not acquired but it adds exceeding 3.0wt%, it is not desirable.

[0008] A alloy and B alloy perform coarse grinding with hydrogen processing, a bantamweight division mill, etc., and use it as coarse powder. Subsequently, equalization mixing of the B alloy coarse powder 1 - 30wt% is carried out with a V shaped rotary mixer, a ball mill, etc. to A alloy coarse powder 99 - 70wt%. Less than [ 1wt% ] at this time, a degree of sintering worsens [ B alloy coarse powder ], and in order that a sintered compact consistency may not go up, sufficient magnet property is not acquired. Moreover, since R becomes excessive in [ than 30wt% ] more, only a small residual magnetic flux density is obtained. the coarse

powder after mixing -- R -- 27 - 31wt% and B -- 0.5 - 2.0wt% and Co -- 0.5 - 5wt% and M -- 0.01 - 1.0wt% -- it is good to make it contain. Since the liquid phase required for sintering less than [ 27wt% ] is not acquired but R of the coarse powder after mixing becomes a low sintered compact consistency, magnetic properties are low. Since R becomes [ R ] excessive by the case more than 31wt(s)%, only a small residual magnetic flux density is obtained. Since R2T14 B phase is not formed enough less than [ 0.5wt% ], but B produces nonmagnetic B rich phase when [ than 2.0wt% ] more, only a residual magnetic flux density with small all is obtained. Less than [ 0.5wt% ] lowers a residual magnetic flux density greatly by the case many, from 5wt% rather than as for the amount of Co(es), it is enough as corrosion resistance and thermal resistance. Furthermore, coercive force sufficient less than [ 0.01wt% ] is not acquired, but since the rate of the volume of R2T14 B phase falls in [ than 1.0wt% ] more, as for M, a residual magnetic flux density falls.

[0009] Thus, with grinders, such as a jet mill, the amount of content oxygen pulverizes in a 0wt% inert gas ambient atmosphere substantially, and uses raw material coarse powder which mixed obtained A alloy coarse powder and B alloy coarse powder as fines with a mean particle diameter (based on an air permeability method) of about 2-10 micrometers. In recovery of fines, the container which filled the special solvent which can prevent advance of oxidation of fines, such as straight mineral oil, vegetable oil, and synthetic oil, to fines recovery openings, such as a jet mill, is installed, and direct fines are collected and slurred in the special solvent in an inert gas ambient atmosphere. In this way, a wet compaction is carried out all over a magnetic field using the metal mold to which the raw material of the shape of an acquired slurry is made as for grant of a predetermined magnetic field anisotropy, and it considers as a Plastic solid. Since said impalpable powder particle is held in a special solvent in the state of a slurry or a Plastic solid and it is isolated with oxygen, the amount of oxygen of fines or a Plastic solid does not increase. Next, this Plastic solid is heated at about 100-300 degrees C with the vacuum furnace of  $1 \times 10^{-2}$  or more torrs,

and the content solvent within a Plastic solid is fully removed. Subsequently, succeedingly, the temperature of the same vacuum furnace is pulled up to about 1000-1200 degrees C, for example, it sinters under the degree of vacuum of  $1 \times 10^{-3}$  -  $9 \times 10^{-6}$  torr. The sintered compact at the temperature of under sintering temperature subsequently, for example, the rare earth sintered magnet of this invention obtained by carrying out heat treatment of two steps or a three-stage R:27.0 - 31.0wt%, and B:0.8 - 1.02wt % and Co:0.5 - 5.0wt%, M:0.01 - 1.0wt%, 0.02 - 0.15wt%, a content carbon content has less than [ 0.15wt% ], and the remainder has [ the amount of content oxygen / less than / 0.25wt% / and content nitrogen volume ] the presentation of Fe. The amount of R, B, Co, and Fe is based on presentation adjustment of a coarse powder raw material. Although the oxygen to contain, nitrogen, and carbon are contained as an impurity in the raw material alloy or it mixes from atmospheric air, a special solvent, etc. at the time of coarse grinding and heating In order for these unescapable impurities to make nonmagnetic compounds, such as  $\text{Nd}_2\text{O}_3$ ,  $\text{Nd}_2\text{C}_3$ , and  $\text{NdN}$ , in a R-T-B system rare earth magnet alloy, to lower the rate of the main phase volume, to decrease the amount of R which works as the liquid phase further at the time of sintering and to check sintering, In the amount of content oxygen, less than [ 0.25wt% ] and content nitrogen volume must make a content carbon content less than [ 0.15wt% ] 0.02 - 0.15wt%. When it is in this presentation range, a R-T-B system rare earth sintered magnet with the high magnetic properties of this invention can be obtained.

[0010] The reason for having magnetic properties with the expensive rare earth magnet of this invention is explained. The oxidation after pulverizing can be prevented by performing wet recovery and a wet compaction in this invention. For example, for example, the amount of content oxygen pulverizes substantially the R-T-B system rare earth magnet of this invention in 0% of inert gas, and said fines are further collected directly in an inert gas ambient atmosphere in the special solvent which can prevent oxidation of fines. Furthermore, under coexistence of this special solvent, it can fabricate among a magnetic field, and

the amount of content oxygen, content nitrogen volume, and a content carbon content can be stabilized and held down to the above-mentioned range by fully carrying out the deliquoring afterbaking join of the acquired Plastic solid. Since a presentation and organization of A alloy and B alloy were furthermore set up as optimal thing to this hypoxia method, the degree of sintering etc. is improvable, and even if it does not add the nonmagnetic element which prevents grain growth of Nb currently conventionally used abundantly, as a result of being able to make the organization in a rare earth magnet alloy into the best thing, it became possible to raise the detailed rate of the main phase ratio compared with the former. Furthermore, alloying elements, such as Co and Dy, are added into B alloy, and distribution of Dy element in the main phase crystal grain can be controlled now. That is, it became possible to obtain the R-T-B system rare earth magnet alloy which was excellent in magnetic properties with the amount of hypoxia by pulling out enough the potential which the blending method has by the hypoxia method. in addition -- the case of the jet mill grinder of the production mold which has the capacity that for example, R-Fe-B system raw material coarse powder can be pulverized with 10 kg/Hr extent, with the inert gas whose above-mentioned oxygen density is 0wt% substantially -- the oxygen density in inert gas -- less than [ 0.01vol% ] -- more -- desirable -- less than [ 0.005vol% ] -- the inert gas not more than 0.002vol% is said especially preferably.

[0011]

[Embodiment of the Invention] Hereafter, although this invention is concretely explained with an example, the contents of this invention are not limited by this.

[0012] (Example 1) In order to produce the alloy A which consists of Ga0.1%, Cu0.1%, and the remainder Fe Nd27.5%, Pr0.4%, and B1.05% with weight percent, specified quantity weighing capacity of Nd, Pr, B, Ga, Cu, and Fe of 95% or more of purity was carried out, the RF dissolution was carried out in Ar gas ambient atmosphere, similarly teeming of this alloy molten metal was carried out to the copper single roll in Ar gas ambient atmosphere, and it considered as the sheet metal-like alloy by the so-called strip cast method. The board thickness

of this alloy was 0.1-0.3mm. Furthermore, Alloy A was inserted in all over the vacuum furnace, and heat treatment was carried out under the conditions of  $5 - 8 \times 10^{-2}$  Torr for 1000 degree-Cx 4 hours. Moreover, in order to produce the alloy B which consists of Nd31.5%, Pr0.5%, Dy15.0%, aluminum0.2%, Co20.0%, and the remainder Fe with weight percent, in Ar gas ambient atmosphere, the RF dissolution was carried out and Nd, Pr, Dy, aluminum, Co, and Fe of 95% or more of purity were cast. Thus, after heating the obtained alloy A and Alloy B to 500 degrees C, performing evacuation after carrying out occlusion of the hydrogen in the furnace which eliminated air, it cooled, and coarse grinding of them was carried out after that, and they were used as the coarse powder of a 32-mesh undershirt. Next, to the coarse powder 90 weight section of Alloy A, in the after [ weighing capacity ] V shaped rotary mixer, equalization mixing was carried out and the coarse powder 10 weight section of Alloy B was used as raw material coarse powder. It inserted in in the jet mill which carried out N<sub>2</sub> inert gas replacement of this raw material coarse powder, and made the oxygen density 0.001vol(s)%, and jet mill grinding was carried out. The container which filled straight mineral oil (the Idemitsu Kosan make, trade name Idemitsu super sol PA-30) was installed in grinding fines recovery opening of a jet mill, and fines were collected and slurred in direct straight mineral oil in N<sub>2</sub> gas ambient atmosphere. The mean particle diameter (air permeability method) of fines was 4.5 micrometers. Next, this slurry was poured in impressing the magnetic field of 10.5kOe(s) in the mold cavity of predetermined magnetic field metal mold, and the wet compaction was carried out with the moulding pressure of 1.0 ton/cm<sup>2</sup>. The impression approach of an orientation magnetic field is perpendicular to the shaping direction. Next, after heating the acquired Plastic solid in the vacuum of  $5 - 7 \times 10^{-2}$  torr for 200 degree-Cx 1 hour and fully removing content straight mineral oil, the temperature up was continuously carried out to 1070 degrees C with 15-degree-C programming rate for /in the vacuum of  $5 - 8 \times 10^{-5}$  torr with the same vacuum furnace, and at the temperature, it held for 2 hours, sintered and cooled. Next, the line obtained the rare earth magnet of this invention for heat

treatment of 900 degree-Cx 2 hours, and 480 degree-Cx 1 hour once each in Ar ambient atmosphere to this sintered compact. The typical presentations of this rare earth magnet were Co2.1%, aluminum0.1%, Ga0.1%, Cu0.1%, the 0.17% of the amounts of content oxygen, 0.05% of content nitrogen volume, 0.07% of content carbon contents, and Remainder Fe Nd27.7%, Pr0.6%, Dy1.5%, and B0.92% in weight percent. Moreover, the typical magnetic properties in 20 degrees C of this rare earth magnet are the good magnetic properties of  $B_r=14.2\text{kG}$ ,  $iH_c=14.9\text{kOe}$ , and  $(BH)_{\text{max}}=48.4\text{MGOe}$  as they are shown in Table 1. Moreover, although gazed at the microstructure of the thing of the arbitration of the obtained rare earth magnet with the optical microscope, B rich phase was not observed. moreover, every which is in the size range shown on the axis of abscissa of drawing 1 to the gross area of the observed R2T14B main phase crystal grain of a visual field -- the rare earth magnet obtained in this example 1 for the axis of ordinate of drawing 1 in the rate of area (%) which <sup>\*\*</sup>(ed) area of the R2T14B main phase with that gross area was evaluated. Here, 16-17 of the particle size (micrometer) which is for the axis of abscissa of drawing 1 show the R2T14B main phase which whose diameter of crystal grain is 16 micrometers or more, and is in the range of less than 17 micrometers. Moreover, the diameter of crystal grain of the R2T14B main phase in drawing 1 and the rate of area were measured as follows, and were evaluated. First, using the optical microscope of form NIKON UFX-II, the optical microscope photograph (one 1000 times the scale factor of this) of the arbitration part of the above-mentioned micro observation sample was taken, it read from the scanner with which the predetermined personal computer with which this optical microscope photograph is subsequently installed in the image-processing software (Image pro.plus (DOS/V)) by the PURANE TRON company was equipped, and the image processing was carried out. the area of each <sup>\*\*\*\*</sup> crystal grain of the arbitration measured by this image processing -- (Si) -- carrying out -- further -- the observation cross-section configuration of each <sup>\*\*\*\*</sup> crystal grain -- a circle -- assuming -- each -- it defined by diameter (di)=of <sup>\*\*\*\*</sup> crystal grain  $(4 \times Si/\pi)^{1/2}$ .

Moreover, the ratio of the area ( $S_i$ ) of the main phase crystal grain in the size range shown on the axis of abscissa of drawing 1 to the whole surface product ( $S_{total}$ ) of the observed target main phase crystal grain, i.e., (rate of area),  $=S_i/S_{total} \times 100(\%)$ , defined the rate of area which is the main phase. As an evaluation result was shown in drawing 1, the rate of area of the crystal grain of 16 micrometers or more of diameters of crystal grain of the rate of area of the crystal grain of less than 14 micrometers of diameters [ in / in the thing of an example 1 / the R2T14B main phase ] of crystal grain was 4.0% 83.2%.

Moreover, the result of having analyzed Fe, Nd and Dy of the thing of the arbitration of the obtained rare earth magnet, and the behavior of B content element by EPMA is shown in drawing 2. Although between the maximum peaks of Nd is the main phase crystal grain in drawing 2, it turns out that inclination is in Dy concentration in this main phase crystal grain. Near the grain boundary of the main phase crystal grain, Dy concentration is high and is in the inclination for Dy concentration to be low, in the core of the main phase crystal grain. This is considered to be because for Dy to have been added only into B alloy. What the core of the main phase crystal grain bears high Br, and is bearing iHc with near [ high ] the grain boundary of the main phase crystal grain by the maldistribution of this Dy distribution is presumed.

[0013] (Example 2) The R-T-B system anisotropy sintering rare earth magnet of this invention was manufactured like the example 1 Nd27.5%, Pr0.4%, and B1.11% with weight percent except having used the alloy A which consists of Ga0.1%, Cu0.1%, and the remainder Fe. The typical component-analysis values of the obtained rare earth magnet were Co2.2%, aluminum0.1%, Ga0.1%, Cu0.1%, the 0.20% of the amounts of content oxygen, 0.04% of content nitrogen volume, 0.07% of content carbon contents, and Remainder Fe Nd27.8%, Pr0.5%, Dy1.5%, and B1.00% in weight percent. Moreover, when the typical magnetic properties of the obtained rare earth magnet were measured in 20 degrees C, as shown in Table 1, the good magnetic properties of Br=14.1kG, iHc=15.0kOe, and (BH) max=47.9MGOe were acquired. Moreover, although that B rich phase

exists slightly has checked with the optical microscope when gazed at the microstructure of the thing of the arbitration of the rare earth magnet of this example 2, the rate of area of B rich phase to the gross area of an observation side was as small as 0.36%.

[0014] (Example 1 of a comparison) The R-T-B system anisotropy sintered magnet was produced like the example 1 Nd27.5%, Pr0.4%, and B1.17% with weight percent except having used the alloy B which consists of Nd31.5%, Pr0.5%, Dy15.0%, aluminum0.2%, Co20.0%, and the remainder Fe with the alloy A which consists of Ga0.1%, Cu0.1%, and the remainder Fe, and weight percent. The typical presentations of the obtained rare earth magnet are Nd27.9%, Pr0.5%, Dy1.5%, B1.08%, and Co2.1%, aluminum0.1%, Ga0.1%, Cu0.1%, the 0.19% of the amounts of content oxygen, 0.05% of content nitrogen volume, 0.07% of content carbon contents, and Remainder Fe in weight percent, and are high compared with examples 1 and 2. [ of B content ] When the typical magnetic properties of the obtained rare earth magnet were measured in 20 degrees C, as shown in Table 1, it was set to  $B_r=13.9\text{kG}$ ,  $iH_c=15.2\text{kOe}$ , and  $(BH)_{\text{max}}=46.9\text{MGOe}$ , and  $B_r$  and  $(BH)_{\text{max}}$  were lower than examples 1 and 2. Moreover, when the microstructure of the thing of the arbitration of the obtained rare earth magnet is evaluated like an example 2, the rate of area of B rich phase is 0.77%, and is twice [ about ] the example 2. Since many nonmagnetic B rich phases were formed since there were many B contents compared with examples 1 and 2, and the rate of the volume of the R2T14B main phase fell relatively from the above thing, the thing of the example 1 of a comparison is considered that  $B_r$  and  $(BH)_{\text{max}}$  are falling.

[0015] (Example 2 of a comparison) The rare earth magnet alloy of the same presentation as the example 1 of a comparison was produced by the single method. Specified quantity weighing capacity of Nd, Pr, Dy, B, aluminum, Ga, Cu, Co, and Fe of 95% or more of purity was carried out, and Co2.0%, aluminum0.15%, Ga0.08%, Cu0.10%, and the raw material alloy that has the presentation of Remainder Fe were obtained Nd27.9%, Pr0.45%, Dy1.5%, and



B1.05% with weight percent by the same strip cast method as an example 1. Thus, after heating the obtained raw material alloy to 500 degrees C, performing evacuation after carrying out occlusion of the hydrogen in the furnace which eliminated air, it cooled, and coarse grinding of it was carried out after that, and it was used as the raw material coarse powder of a 32-mesh undershirt. It inserted in in the jet mill which permuted this raw material coarse powder by N2 gas, and made the oxygen density 0.001vol(s)%, jet mill grinding was carried out, the container which filled straight mineral oil (the Idemitsu Kosan make, trade name Idemitsu super sol PA-30) was installed in fines recovery opening, and in straight mineral oil, the fines which carried out jet mill grinding in N2 gas ambient atmosphere were collected directly, and were slurred. The mean particle diameter (air permeability method) of fines was 4.4 micrometers. The R-T-B system anisotropy sintered magnet was obtained like the example 1 henceforth. The typical component-analysis values of this thing were Co2.0%, aluminum0.1%, Ga0.1%, Cu0.1%, the 0.16% of the amounts of content oxygen, 0.05% of content nitrogen volume, 0.07% of content carbon contents, and Remainder Fe Nd27.7%, Pr0.5%, Dy1.5%, and B1.06% in weight percent. Moreover, when the magnetic properties of the obtained above-mentioned rare earth magnet were measured in 20 degrees C, as shown in Table 1, it is  $B_r=13.8\text{kG}$ ,  $iH_c=14.1\text{kOe}$ , and  $(BH)_{\text{max}}=46.4\text{MGOe}$ , and low magnetic properties were acquired compared with examples 1 and 2. Moreover, the thing of arbitration was chosen from the obtained rare earth magnet, and the result of having evaluated the rate of area of the main phase crystal grain which is in each diameter range of crystal grain like an example 1 was shown in drawing 3. From drawing 3, especially the diameter distribution of crystal grain of the main phase was broadcloth at the diameter side of a large drop compared with drawing 1 (example 1), and the rate of area of the main phase crystal grain beyond particle-size 19micrometer of the rate of area of the main phase crystal grain with a particle size of less than 16 micrometers was 6.7% 83.6% (therefore, 16 micrometers or more 16.4%). Thus, by the single method, since it is easy to

make the main phase crystal grain big and rough and it serves as particle size distribution with it compared with what is depended on the method of blending above-mentioned this invention, it is judged with the single method of control of the main phase grain growth being inadequate, and magnetic properties having fallen rather than the thing of this invention produced by the blending method.

[ large dispersion in the diameter of crystal grain of the main phase, and ]  
[ broadcloth ]

[0016] (Example 3 of a comparison) The R-T-B system rare earth magnet alloy of Nb addition was produced by the single method. Specified quantity weighing capacity of Nd, Pr, B, Nb, Ga, Cu, Co, and Fe of 95% or more of purity was carried out, and Co2.0%, Nb0.70%, aluminum0.15%, Ga0.08%, Cu0.10%, and the raw material alloy that has the presentation of Remainder Fe were obtained Nd27.9%, Pr0.45%, Dy1.5%, and B0.90% with weight percent by the same strip cast method as an example 1. Nb is added in order to control grain growth into this alloy. Grinding, wet recovery, a wet compaction, deliquoring, sintering, and heat treatment were performed like the example 1, and the R-T-B system rare earth sintered magnet of Nb addition was produced henceforth. The typical component-analysis values of this magnet were Co2.0%, Nb0.72%, aluminum0.10%, Ga0.07%, Cu0.12%, the 0.13% of the amounts of content oxygen, 0.08% of content nitrogen volume, 0.07% of content carbon contents, and Remainder Fe Nd27.7%, Pr0.57%, Dy1.5%, and B0.87% in weight percent. Moreover, the magnetic properties in typical 20 degrees C of the obtained rare earth magnet were  $B_r=13.3\text{kG}$ ,  $iH_c=16.1\text{kOe}$ , and  $(BH)_{\text{max}}=42.7\text{MGOe}$  as they were shown in Table 1, and they were lower than examples 1 and 2.

[0017] (Example 4 of a comparison) The R-T-B system rare earth magnet alloy of Nb addition was produced by the single method. Specified quantity weighing capacity of Nd, Pr, B, Nb, Ga, Cu, Co, and Fe of 95% or more of purity was carried out, and Co2.0%, Nb0.70%, aluminum0.15%, Ga0.08%, Cu0.10%, and the raw material alloy that has the presentation of Remainder Fe were obtained Nd27.9%, Pr0.45%, Dy1.5%, and B1.10% with weight percent by the strip cast

method of the same conditions as an example 1. Nb is added in order to control grain growth into this alloy. Grinding, wet recovery, a wet compaction, deliquoring, sintering, and heat treatment were performed like the example 1, and the R-T-B system rare earth sintered magnet was produced henceforth. The typical component-analysis values of this magnet were Co2.1%, Nb0.71%, aluminum0.14%, Ga0.07%, Cu0.12%, the 0.12% of the amounts of content oxygen, 0.06% of content nitrogen volume, 0.08% of content carbon contents, and Remainder Fe Nd27.8%, Pr0.57%, Dy1.5%, and B1.03% in weight percent. Moreover, the typical magnetic properties of the obtained rare earth magnet are Br=13.2kG, iHc=16.5kOe, and (BH) max=42.0MGOe in 20 degrees C, and are low magnetic properties from examples 1 and 2 as they are shown in Table 1. The result of the examples 3 and 4 of a comparison showed that Br was not greatly dependent on the amount of B in the R-T-B system rare earth sintered magnet of Nb addition produced by the single method. Moreover, the result of having analyzed the behavior of Fe, Nd, Dy, and B which are contained about the thing of the arbitration of the rare earth magnet obtained in this example 4 of a comparison by EPMA is shown in drawing 4 . In drawing 4 , the concentration gradient of Dy in the main phase crystal grain which was observed by drawing 1 is not seen. It was checked that the concentration gradient of Dy observed in the example 1 from this is peculiar to blend material.

[0018] The magnetic properties acquired in each above-mentioned example and each example of a comparison were summarized in Table 1.

[Table 1]

	Br(kG)	iHc(kOe)	(BH) <sub>m</sub> (MGOe)
実施例1	14.2	14.9	48.4
実施例2	14.1	15.0	47.9
比較例1	13.9	15.2	46.9
比較例2	13.8	14.1	46.4
比較例3	13.3	16.1	42.7
比較例4	13.2	16.5	42.0

[0019] The rate of area of B rich phase obtained in examples 1 and 2 and the example 1 of a comparison was shown in Table 2.

[Table 2]

	Bリッチ相の面積率(%)
実施例1	0.00
実施例2	0.36
比較例1	0.77

[0020]

[Effect of the Invention] As above-mentioned, while the rare earth magnet of this invention reduced the nonmagnetic phase of oxide and B rich equality compared with the former and raised the rate of the main phase volume, Br and (BH) max were able to be brought close to the potential of a R-T-B system magnet alloy proper by having controlled the grain growth of the main phase. Furthermore, since Nb additive-free can carry out [ detailed ]-izing of the diameter distribution of crystal grain of the main phase, the rate of the volume of a nonmagnetic phase can be reduced more. Thus, while combining a wet process with features and the blending method, the usefulness of this invention considered as the R-T-B system rare earth magnet alloy presentation suitable for the combination is clear.

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[Translation done.]

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## DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] It is drawing showing the relation between the diameter distribution of the main phase crystal grain of the rare earth magnet of this invention, and the rate of area.

[Drawing 2] It is drawing showing the EPMA analysis result of the rare earth magnet of this invention.

[Drawing 3] It is drawing showing the relation between the diameter distribution of the main phase crystal grain of the rare earth magnet of the example of a comparison, and the rate of area.

[Drawing 4] It is drawing showing the EPMA analysis result of the rare earth magnet of the example of a comparison.

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[Translation done.]

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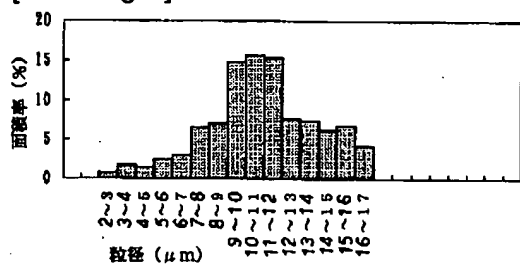
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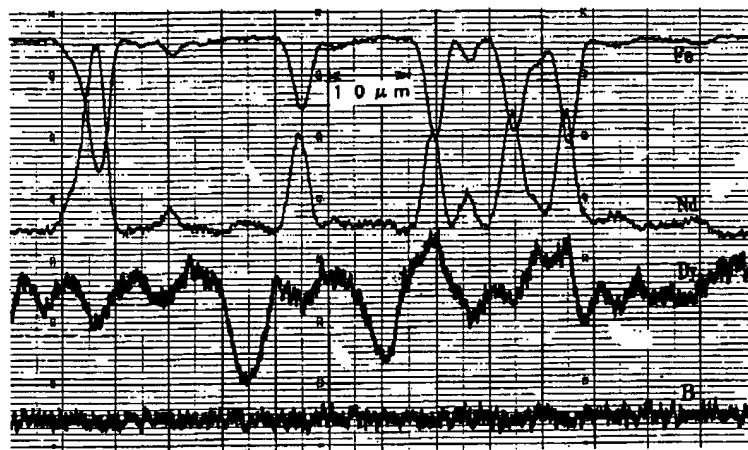
## DRAWINGS

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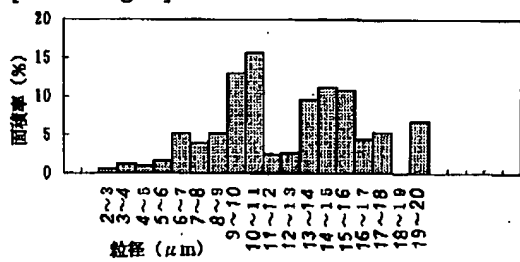
[Drawing 1]



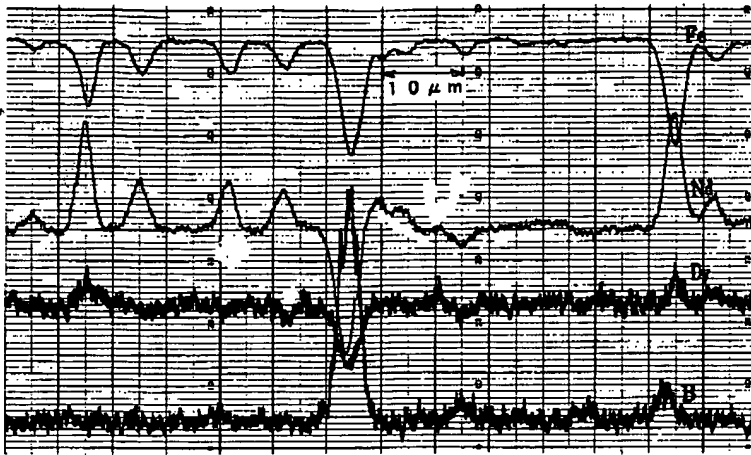
[Drawing 2]



[Drawing 3]



[Drawing 4]



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[Translation done.]